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IS 3025-59 (2006): Methods of Sampling and test (physical and Chemicals) for water and wastewater, Part 59: Manganese [CHD 32: Environmental Protection and Waste Management]



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भारतीय मानक
जल एवं अपशिष्ट जल के नमूने लेना और परीक्षण
(भौतिक एवं रासायनिक) की विधियाँ
भाग 59 मैंगनीज़
(पहला पुनरीक्षण)

Indian Standard
METHODS OF SAMPLING AND TEST (PHYSICAL AND
CHEMICAL) FOR WATER AND WASTEWATER
PART 59 MANGANESE
(*First Revision*)

ICS 13.060.50

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Part 59) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Manganese is a naturally occurring element that is found in rock, soil, water and food. It usually occurs together with iron. Surface water (rivers and reservoirs) do not usually contain high concentrations of manganese because the oxygen rich water enables the mineral to settle out as sediments often associated with micro-organisms and as complexes with, for example, humic acid. In deep wells and springs where the oxygen content and *pH* tend to be low, all manganese will be present in dissolved forms and water containing manganese appears colourless.

Manganese can affect the flavour and colour of water and can also react with tannins present in beverages to form a black sludge affecting both the taste and appearance.

As per IS 10500 : 1991 'Drinking water — Specification (*first revision*)', the desirable limit and permissible limit (in the absence of alternate source) for manganese in the drinking water is 0.1 mg/l, maximum and 0.3 mg/l, maximum respectively. Beyond this level both the taste and appearance are affected and has adverse effect on domestic uses and water supply structure.

The Committee responsible for the formulation of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' had decided to revise the standard and publish it as separate parts. This standard is one of the different parts under the IS 3025 series of standards and supersede 35 of IS 3025.

Formaldoxime method given in this standard is prepared based on the method specified in ISO 6333 : 1986 'Water quality – Determination of manganese – Formaldoxime spectroscopic method'. It is technically equivalent to ISO 6333 : 1986.

The composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER****PART 59 MANGANESE***(First Revision)***1 SCOPE**

This standard (Part 59) prescribes two methods of test for determination of manganese in water and wastewater.

- a) Periodate colorimetric method, and
- b) Formaldoxime photometric method.

2 REFERENCES

The standards listed below contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
3025 (Part 1) : 1986	Methods of sampling and test (physical and chemical) for water and wastewater: Part 1 Sampling (<i>first revision</i>)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 3025 (Part 1). Collect the sample in a polyethylene, polyvinyl chloride or glass container and acidify the sample with sulphuric acid (*see 6.4.9*) until the pH is approximately, but not less than, 1. This acidification minimizes adsorption of manganese on the walls of the container and also assists in the dissolution of colloidal and particulate forms of manganese.

5 PERIODATE COLORIMETRIC METHOD**5.1 Principle**

The manganese in the sample is oxidized with potassium periodate. The pink colour obtained is matched against that produced with a series of standard manganese solutions.

5.2 Range and Applicability

The method is applicable up to 0.2 mg of manganese (as Mn).

5.3 Interference

No information available.

5.4 Apparatus**5.4.1 Nessler Tubes** — 50 ml capacity.**5.5 Reagents****5.5.1 Dilute Sulphuric Acid** — 1:1 v/v.

5.5.2 Hydrogen Peroxide-Nitric Acid Mixture — Mix equal volumes of hydrogen peroxide (30 percent) and concentrated nitric acid. Prepare fresh every day.

5.5.3 Stabilized Distilled Water — Assemble a distillation apparatus consisting of a round bottom 1 litre flask fitted with an efficient splash head, connected to a Liebig condenser by means of ground-glass joints. Introduce 500 to 600 ml of distilled water into the flask, add about 0.1 g of potassium permanganate previously dissolved in 5 ml of water and a few drops of dilute sulphuric acid. Distill carefully taking care that there is no carryover by splashing or otherwise of liquid from the flask, and reject the first 50 ml of distillate.

5.5.4 Phosphoric Acid — Specific gravity 1.75.

5.5.5 Potassium Periodate

5.5.6 Standard Manganese Solution — Measure 45.5 ml of 0.1N potassium permanganate solution into a 250-ml

beaker, add a few drop of dilute sulphuric acid, heat to boiling and then add a saturated solution of sulphur dioxide in water drop by drop until the permanganate is just decolourized. Boil for 15 min, cool, transfer the solution to a 500-ml graduated flask, dilute to the mark and mix well. Measure 100 ml of the solution into a 500-ml graduated flask, add 5 ml of dilute sulphuric acid, dilute to the mark and mix well. One millilitre of the dilute solution is equivalent to 0.02 mg of manganese (as Mn).

5.6 Procedure

To a 300-ml conical flask of borosilicate glass measure a suitable volume of the well mixed sample (*see* Note). Add 4.0 ml of dilute sulphuric acid and evaporate to fuming. Whilst heating, treat with the hydrogen peroxide-nitric acid mixture, adding a few drops at a time, until all traces of organic matter are completely destroyed. Cool, add 10 ml of stabilized distilled water and evaporate to fuming; again cool and repeat the addition of water followed by evaporation. Add 50 ml of stabilized distilled water, 2 ml of the phosphoric acid and 0.2 g of potassium periodate, bring to the boil and keep just below the boiling point for 1 hour. Cool to room temperature, transfer the solution to a Nessler tube, adjust the volume to 50 ml with stabilized distilled water and mix well. Into seven 300-ml conical flasks measure by means of a burette, 0, 1.0, 2.0, 4.0, 6.0, 8.0 and 10 ml of standard manganese solution. Treat as described above for the sample. Transfer the solutions to Nessler tubes, dilute to the mark with stabilized distilled water and mix. Compare the colours of the sample and standards.

NOTE — Suitable volumes of sample are as follows:

Sl No.	Calcium Content of Sample As CaCO ₃ mg/l	Volume of Sample ml
(1)	(2)	(3)
i)	Up to and including 250	200
ii)	251-500	100
iii)	501-1 000	50

5.7 Calculation

$$\text{Manganese (as Mn), mg/l} = 1\,000 \frac{W}{V}$$

where

W = amount of manganese present in the standard which matches the colour obtained with the sample, in mg; and

V = volume of the sample taken for the test, in ml.

6 FORMALDOXIME SPECTROMETRIC METHOD

6.1 Range and Applicability

The method is applicable to the determination of

manganese concentrations between 0.01 mg/l and 5 mg/l. Manganese concentrations above 5 mg/l may be determined after suitable dilution of the sample.

NOTE — This method is not applicable to highly contaminated waters such as industrial wastewater.

6.2 Principle

Manganese reacts with formaldoxime solution to form an orange-red complex. The manganese formaldoxime complex is stable between pH values of 9.5 and 10.5 and the intensity of the colour produced is proportional to the amount of manganese present. Manganese is determined spectrometrically at a wavelength of about 450 nm.

NOTE — If suspended or organically bound manganese is present, pre-treatment is required to convert manganese to forms capable of reacting with formaldoxime.

6.3 Apparatus

6.3.1 Spectrometer, with selectors for continuous variation (prism or grating type) or discontinuous variation (narrow band pass optical filter) capable of measuring absorbance at approximately 450 nm equipped with cells of optical path lengths up to 100 mm (for manganese concentrations of less than 0.3 mg/l) and 10 mm (for manganese concentrations above 0.3 mg/l).

6.3.2 Glass Bottle, capacity 100 ml, provided with ground glass stoppers and metal clamps or with screw-caps of non-coloured plastic, suitable for autoclaving.

NOTE — All glassware and sampling containers shall be washed with approximately 1 mol/l hydrochloric acid (HCl) and rinsed with water before use.

6.3.3 Autoclave or Pressure Cooker, capable of maintaining a temperature of 120°C and a pressure of 200 kPa.

6.4 Reagents

6.4.0 Purity of the Reagents — Unless specified otherwise, only pure chemicals and fluoride free distilled water shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

6.4.1 Oxidizing Reagent, either potassium peroxodisulphate (K₂S₂O₈) or sodium peroxodisulphate (Na₂S₂O₈).

6.4.2 Sodium Sulphite (Na₂SO₃), anhydrous.

6.4.3 EDTA, Tetrasodium Salt, Solution, c(EDTA) = 0.24 mol/l — Dissolve 90 g of disodium ethylenedinitrilotetra-acetic acid (Na₂EDTA) dihydrate

($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$) and 19 g of sodium hydroxide (NaOH) in water and dilute to 1 000 ml.

Alternatively, dissolve 109 g of tetrasodium ethylenedinitrilotetra-acetic acid (Na_4EDTA) tetra hydrate ($C_{10}H_{12}N_2Na_4O_8 \cdot 4H_2O$) or 100 g of tetrasodium ethylenedinitrilotetra-acetic acid dihydrate ($C_{10}H_{12}N_2Na_4O_8 \cdot 2H_2O$) in water and dilute to 1 000 ml.

6.4.4 Formaldoxime Solution — Dissolve 10 g of hydroxylammonium chloride (NH_2OHCl) in about 50 ml of water. Add 5 ml of 35 percent (*m/m*) methanal ($HCHO$) (formaldehyde) solution ($c = 1.08$ g/ml) and dilute with water to 100 ml.

NOTE — Keep the bottle in a dark and cool place. The solution has a shelf-life of at least 1 month.

WARNING — The reagents described in 6.4.4 and 6.4.6 should be regarded as special hazards. Hazardous operations should be carried out in a fume cupboard. Care must be taken to avoid ingestion or inhalation of vapours and to protect the hands, eyes and face. Gloves and goggles must be worn and any suspected skin contamination washed off immediately. Inhalation of the vapours of formaldehyde and formaldoxime will result in severe irritation and oedema of the upper respiratory tract.

6.4.5 Hydroxylammonium Chloride/Ammonia Solution

6.4.6 Hydroxylammonium Chloride Solution ($NH_2OHCl = 6$ mol/l) — Dissolve 42 g of hydroxylammonium chloride in water and dilute to 100 ml.

6.4.7 Ammonia Solution ($NH_3 = 4.7$ mol/l) — Dilute 70 ml of concentrated ammonia ($c = 0.91$ g/ml) with water to 200 ml.

6.4.8 Ammonium Iron (II) Sulphate Hexahydrate Solution [$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$] — 700 mg/l.

6.4.9 Sulphuric Acid ($H_2SO_4 = 3$ mol/l) — Carefully add 170 ml of concentrated sulphuric acid ($\rho = 1.84$ g/ml) to 750 ml of water. Allow to cool and dilute to 1 000 ml. This solution is commercially available H_2SO_4 ($\rho = 1.19$ g/ml).

6.4.10 Working Ammonium Iron (II) Sulphate Hexahydrate Solution — Dissolve 700 mg of ammonium iron(II) sulphate hexahydrate in water, add 1 ml of sulphuric acid (see 6.4.9) and dilute to 1 000 ml.

6.4.11 Sodium Hydroxide Solution ($NaOH = 4$ mol/l) — Dissolve 160 g of sodium hydroxide in water and dilute to 1 000 ml.

6.4.12 Manganese Standard Solution, Corresponding to 100 mg of Mn per litre — Dissolve 308 mg of manganese sulphate monohydrate ($MnSO_4 \cdot H_2O$) in water in a 1 000 ml one-mark volumetric flask. Add 10 ml of sulphuric acid (see 6.4.9), make up to the mark with water and mix. One ml of this standard solution contains 0.1 mg of Mn.

NOTE — Commercially available standard solutions can also be used.

6.5 Procedure

6.5.1 Test Portion

Take as the test portion 50 ml, or an accurately measured aliquot diluted to 50 ml, of the acidified test sample containing less than 0.25 mg of manganese (5 mg/l).

6.5.2 Preparation of Test Solution

If organically bound or suspended manganese is present, add 225 ± 25 mg of oxidizing reagent (see 6.4.1) to the test portion (see 6.5.1). The oxidation can be performed in one of two ways:

- autoclave the mixture for 30 min in a bottle (see 6.3.2); cool and add approximately 0.5 g of sodium sulphite (see 6.4.2) to reduce oxidizing substances; and
- boil the mixture in a 100 ml conical flask or beaker for about 40 min; cool and transfer the mixture to a 50 ml one-mark volumetric flask, make up to the mark with water and add approximately 0.5 g of sodium sulphite (see 6.4.2) to reduce oxidizing substances. Autoclaving is preferable in the case of samples containing humic acids. If the procedure is not to be continued immediately, the pre-treated sample may be kept overnight.

NOTE — Any turbidity and colour is destroyed during the pre-treatment. If experience has shown that this pre-treatment stage is not required, for example, in most cases for drinking water, it may be omitted.

6.6 Blank Test

Carry out a blank test in parallel with the determination, replacing the test portion by 50 ml of water. If the absorbance of the blank test differs significantly from the extrapolated absorbance of the zero member (see 6.7.4), the reasons for this difference shall be investigated.

6.7 Calibration

6.7.1 Preparation of the Set of Calibration Solutions

- Range A: 0 to 0.5 mg/l manganese**

Dilute 5 ± 0.05 ml of standard manganese solution (see 6.4.12) to 1 000 ml with water in a 1 000 ml one-mark volumetric flask. To a series of five 50-ml one-mark volumetric flasks add 0, 10, 20, 30 and 40 ml of this diluted manganese solution and dilute to the mark with water. This gives calibration standard solutions of 0, 0.1, 0.2, 0.3 and 0.4 mg/l of manganese.

- Range B: 0 to 5 mg/l manganese**

Dilute 50 ± 0.5 ml of standard manganese solution (see 6.4.12) to 1 000 ml with water in a 1 000 ml

one-mark volumetric flask. To a series of five 50-ml one-mark volumetric flasks add 0, 10, 20, 30 and 40 ml of this diluted manganese solution and dilute to the mark with water. This gives calibration standard solutions of 0, 1, 2, 3 and 4 mg/l of manganese.

6.7.2 Colour Development

Add 1 ml of ammonium iron (II) sulphate hexahydrate solution (see 6.4.8) and 2 ml of EDTA, tetrasodium salt solution (see 6.4.3) to each of the solutions. After mixing, add 1 ml of the formaldoxime solution (see 6.4.4) and immediately add 2 ml of sodium hydroxide solution (see 6.4.11). Thoroughly mix the solutions and allow to stand for 5 to 10 min, then add, whilst mixing, 3 ml of hydroxylammonium chloride/ammonia solution (see 6.4.5) and leave to stand for at least 1 h.

6.7.3 Spectrometric Measurements

Between 1 h and 4 h after colour development, measure the absorbances of the solutions using the spectrometer at a wavelength of 450 nm against water as reference. For calibration solutions in range A (0 to 0.5 mg/l manganese) use cells of 100 mm optical path length and for range B (0 to 5 mg/l manganese) use cells of 10 mm optical path length.

6.7.4 Plotting the Calibration Graph

For each set of calibration solutions, prepare a calibration graph by plotting the manganese concentration, expressed in milligrams per litre, of the solution as abscissa against the corresponding absorbance as ordinate. It is essential that a linear calibration graph be achieved. The calibration factor, I , is the reciprocal of the slope of the calibration graph. The intercept of the calibration graph on the ordinate gives the extrapolated absorbance of the zero member of the set of calibration solutions. The calibration factor can also be calculated by regression analysis.

6.7.5 Frequency of Calibration

Each graph shall be checked periodically, and especially when new reagents are used, to ensure repeatability.

6.8 Determination

6.8.1 Colour Development

Proceed in accordance with 6.7.2, but using the test solution (see 6.5.2) instead of the calibration solutions. If the test solution has been pre-treated (see 6.5.2), increase the amount of sodium hydroxide solution (see 6.4.11) from 2 to 2.5 ml.

6.8.2 Spectrometric Measurements — See 6.7.3.

6.9 Expression of Results

6.9.1 Calculation

The manganese concentration, expressed in mg of Mn per litre, is given by the formula:

$$\text{Mn} = f(A_1 - A_0)g$$

where

f = calibration factor appropriate to the particular calibration graph chosen and derived as stated in 6.7.4, expressed, in mg/l;

A_1 = absorbance of the test solution (see 6.8.2);

A_0 = extrapolated absorbance of the zero member (see 6.7.4); and

g = a factor given by the formula:

$$g = \frac{V_1}{V_2}$$

where

V_1 = maximum volume, of the test portion (here 50 ml), in millilitre; and

V_2 = volume, of the test portion, if an aliquot was taken, in millilitre.

NOTE — The volume of acid added (see 5) to the sample shall be taken into consideration in the calculation.

Report the results to the nearest 0.01 mg/l for manganese concentrations from 0.01 to 1 mg/l; and to the nearest 0.1 mg/l for manganese concentrations greater than 1 mg/l.

6.10 Precision

See Table 1.

6.11 Interferences

Ferric ions form a violet complex with formaldoxime which interferes with manganese determination. The addition of EDTA (see 6.4.3) and hydroxylammonium chloride/ammonia (see 6.4.5) reduces the interference; however, it has been shown that the best method of overcoming this effect is to add a constant known amount of iron (III) as ammonium iron (II) sulphate to each calibration solution, blank test and test solution. The presence of 1 mg of cobalt (Co) per litre gives a response equivalent to 40 µg of manganese per litre. If calcium is present, orthophosphate concentrations above 2 mg of phosphorus (P) per litre cause low results. The presence of calcium and magnesium in combined concentrations above 300 mg/l causes high results. If turbidity is present after formation of the coloured complex, centrifuge the solution before measurement of the absorbance (see 6.4.3).

Table 1 Reproducibility of the Method
(Data Derived from an Inter Laboratory Trial Carried Out in 1982)
(Clause 6.10)

Sl No.	Manganese Concentration mg/l	Laboratory	Number of Results	Mean Value mg/l	Standard Deviation mg/l
(1)	(2)	(3)	(4)	(5)	(6)
i)	0.050	B	30	0.049	0.003 5
ii)	0.100	A	30	0.12	0.025
iii)	0.100	B	30	0.099	0.002 1
iv)	0.500	B	30	0.497	0.008 5
v)	1.000	A	30	1.01	0.04
vi)	1.000	B	30	1.001	0.009 1
vii)	2.000	A	30	1.99	0.035
viii)	2.000	B	30	2.055	0.011 1
ix)	4.000	A	30	4.02	0.047
x)	4.000	B	30	4.198	0.133 3

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. CHD 32 (1297).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones: 2323 0131, 2323 3375, 2323 9402

website : www.bis.org.in

Regional Offices:

	Telephones
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 2323 7617 2323 3841
Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	{ 2337 8499, 2337 8561 2337 8626, 2337 9120
Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022	{ 260 3843 260 9285
Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113	{ 2254 1216, 2254 1442 2254 2519, 2254 2315
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